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Potentials of separation membranes in the sugar industry

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Abstract

There are described some applications of membrane separation, e.g. reverse osmosis in the treatment of sugar beat press water, recovery of salt from waste brine at a sugar decolorization plant or microfiltration and ultrafiltration of raw sugar juice, as an alternative to chemical purification process. The last case is completed by original experimental work. Samples were treated with cross flow micro- and ultrafiltration on ceramic membranes having porosity 20 and 50 nm. The changes in the content of sucrose, invert sugar and lactic acid were observed, resulting in increasing the juice purity from the initial value 89% to the final value 91–92%. In addition, permeate can be a subject for direct crystallisation after thickening to produce the final product. Retentate purity decreased to 87–88%. For nanofiltration tests (NF) there has been designed a special stainless steel cross-flow dynamic cell for pressures up to 4 MPa. The preliminary NF tests were focused on testing different commercial flat polymeric membranes (Hydronautics-Nitto Denko, Osmonics-Desal). © 2002 Elsevier Science B.V. All rights reserved.

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1. Introduction

Sugar processing is one of the most energy-intensive processes in the food and chemical industries. Therefore, membrane separation processes (MSP) like microfiltration (MF), ultrafiltration (UF), nanofiltration (NF) or reverse osmosis (RO) seems to find several applications there. On the other hand, some limitations exist for application of MSP in the sugar industry, since the volumes pumped are very high comparing to other food industry branches, the solutions exhibit high viscosity and high osmotic pressure. In Fig. 1, there is a simplified scheme of sugar production, with indicated black points of possible application of MSP.

2. Review

2.1. Clarified thin juice RO concentration

Bichsel et al. [1], mentioned already in 1982 that approximately 50% of the energy utilised to produce beet and cane sugar is consumed during the evaporation of water. That is why the highest potential for energy reduction is concentration of clarified thin juice from 12 to 14% of refractometric dry substance (RDS) to a maximum of 30%

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RDS while osmotic pressure is already about 3.5 MPa (See item 1 in the Fig. 1). Even when the author reached in his experiments on non-cellulose acetate polymeric membranes flux in the range from 20 to 10 $1/h/m^2$ with the concentration between 15 and 25% RDS, he concluded that many problems associated with thermal creep within the membrane modules must be further solved. Moreover, higher osmotic pressure of concentrated thin juice needs higher energy inputs, what impedes an easy industrial application.

2.2. Raw juice UF purification

It seems that membrane separations are limited more or less by dilute streams and that is why the main interest is focused on purification of the juices from the extraction stage (See item 2, Fig. 1), where viscosity, dissolved solid concentration and temperature are lower. Hanssens et al. [2] stated already in 1984 that no fouling problems occurred at a tangential velocity of 4 m/s, no pre-filtration was needed during UF clarification of raw juice to the same degree as achieved by the conventional process. But he still recommends further treatment with 0.05% lime, electrodialysis or ion exchange to remove remaining impurities. Mak [3] described in 1991 the removal of colour impurities from raw sugar by ultrafiltration. He applied the filtration unit Alfa-Laval with modules PM10, which are 1.1 m long and filled with hollow fibres. The filtration area is 2.46 m per one module. Proteins, starches, gums, colloids and colour impurities were removed. The filtration of juice prepared from raw sugar was either a single-stage process removing 75% of colour impurities or it involved a recycled mode in which colour level fell by 60–90% (depending on the type of juice).

Some researchers have aimed on conditions of separation processes. Establishing of optimal cross-flow process conditions for microfiltration (CMF) and ultrafiltration (UF) of raw sugar cane juice have been studied by Domier et al. [4,5] in 1993. They also report, that progressively increasing both transmembrane pressure and cross-flow velocity in the initial stage of CMF of raw cane sugar remelts on ceramic membranes Membralox

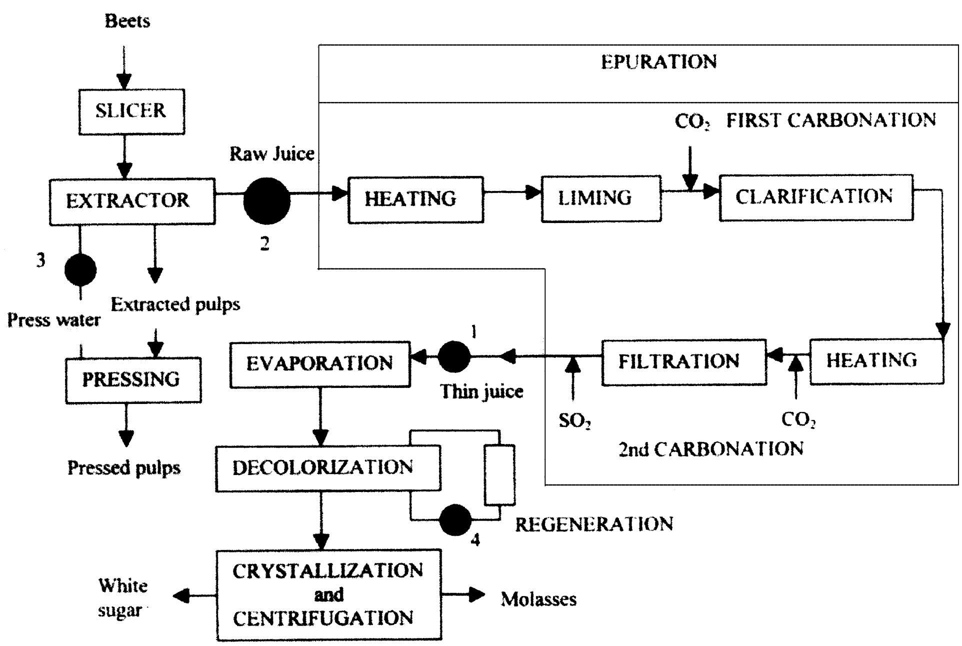

Fig. 1. Simplified scheme of sugar production with indicated spots of potential MSP applications.

1.4 μm at 80°C results in 13–26% improvement of permeate flux comparing to commonly used abrupt start-up procedure. Nevertheless it is mentioned, that in numerous cases, the permeate fluxes usually obtained are still too low to face any industrial application. Various process conditions and factors influencing the effectiveness of CMF are discussed by Mikulášek [6] in 1996. The effect of conducting back flush was discussed by Cakl [7] in 1997. Both authors, however, are not focused specifically on sugar solutions.

Recently the attention was transferred to ceramic membranes. Lancrenon et al.[8] analysed in 1993 the application of microfiltration (the size of pores was 0.1–10 μm) and ultrafiltration (2–200 nm) in sugar beet and sugar cane refinements. The Applexion system with ceramic membranes Carbosep (carbon base) and Kerasep (aluminium oxide or titanium base) is described. During an experimental ultrafiltration of sugar beet raw juice, the permanent output of 200 $l/h/m^2$ was achieved with this filter. The author believes that such a flux, which can be steadily reached by ultrafiltration, brings the process to the point where it should be considered as a very credible alternative to conventional carbonation. The goal of direct production of white cane sugar by means of clarification and decolorization membranes is followed by Saska et al. [9] in his article reporting about concentration and decolorization of dilute products from cane molasses desugarization with RO and NF membranes. Vern et al. [10] mentioned membrane technologies in his vision of future sugar refinement in 1995. By microfiltration of raw juice through the Filmtec Selectflo CMF synthetic membranes with porosity of 0.2 μm, such purity of raw juice was achieved that direct crystallisation was possible without the complex process of purification which involves calcium hydroxide and CO_2 addition, cake filtration, lime kiln etc. The filtration modules are 1 m long, diameter 150 mm. Internal filtration channels have diameter of 3 mm, total filtration area is 5.5 m^2 per one module. The process involved cross-flow filtration followed in individual stages. The content of colour impurities in the final juice was lower (approximately by 80%). The turbidity (the content of colloids) fell to less than 1% of its initial value. The purity increased by 2 points.

Koekoek et al. [11] described in 1997 the nanofiltration experiments with various membranes to examine a possible long-term efficiency in purification of thin juice. The Toray, Desal and Stork membranes were applied. The results did not indicate the possibility of their long-term application in sweetener's technologies. The paper includes great possibilities achieved in the purification of raw sugar juice by ultrafiltration through the ceramic membranes. The preliminary results of the raw juice treatment by microfiltration exactly on ceramic membranes are specified in the papers published by Bubnik et al. [12,13] in 1998 and 1999. By the same authors, there were studied the process of fouling during MF/UF of sugar juice. Mathematical description of the flux decline [13] was presented and it was also found that the increasing temperature of the juice from 30 to 60°C during the process had no significant influence on fouling. The benefit from lower viscosity at elevated temperature was compensated by the thermal decomposition of juice resulting in higher requirements on membrane cleaning [14].

Physico-chemical interactions between particles of mineral membranes (α-Al_2O_3, ZrO_2, TiO_2) and sugar remelts during CMF were studied by Khayat et al. [15] in 1997 with the aim to help to choose the proper membrane depending on the solution to be treated. Vercellotti et al. [16] reported in 1998 the results of his comprehensive analysis of unknown compositional factors of juices or syrups impeding sugar crystallisation, forming evaporator build-ups or drastically changing membrane flux. Preparative membrane techniques utilising a series of different UF, dialysis or MF membranes on ceramic and polymeric base were applied together with other methods like gel chromatography, or nuclear magnetic resonance spectroscopy. The found results were, that the principal responsibility for fouling of UF membranes is on soluble polysaccharides, high molecular weight colorants and other large molecules and colloidal complexes together with fine suspended solid particles. After removing of these components by disc centrifuge, the flux was improved by 60 to 130%. Results indicate that sugar juice pre-filtration prior to the membrane separation can yield in a substantial cost cutting

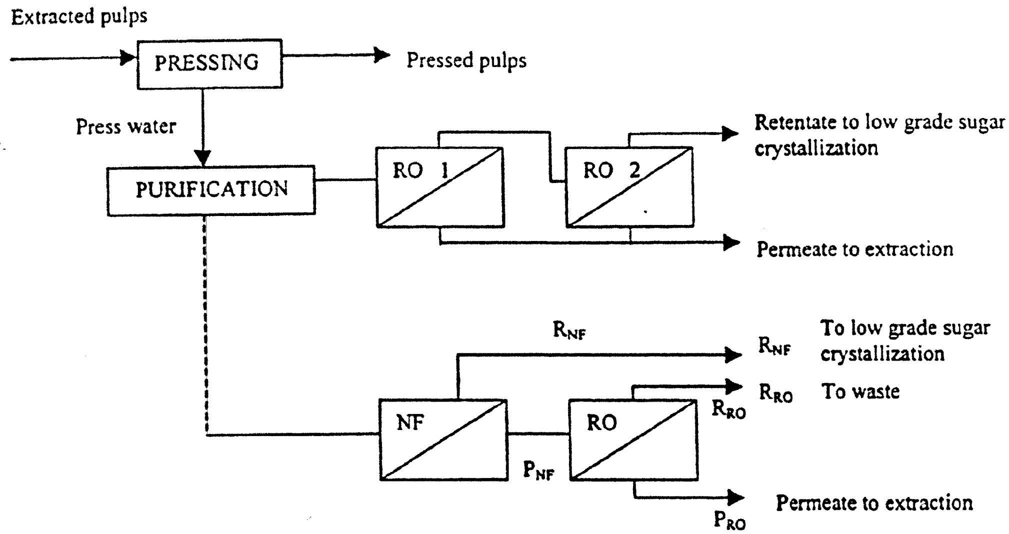

Fig. 2. Schematic of press water treatment with RO.

of subsequent UF/MF. This represents an approach alternative to another conception of UF/MF process applied directly on non-filtered raw juice, where savings were found in the absence of pre-filtration operation.

Some other works dealing with micro- and ultrafiltration of cane and beet sugar raw juices are summarised in Handbook of Cheryan et al. [17].

2.3. Sugar beet press water RO/NF clarification

Another process stream, which was experimentally studied by Bogliolo et al. [18] with regards to MSP, was the stream of press water (See item 3 in the Fig. 1). Nowadays this stream is completely recycled to the sugar extraction unit. Press water derives from the pressing station of the extracted pulp after it passed through the extraction unit. Press water typically contains 1–3% of total solids. The composition of solids is 60–80% of sugars, 20–40% of salts, colloids and suspended impurities. The amount of water is large, about 0.6 kg per kg of beet input, so during the processing of 10 000 ton of beet a day there recycles about 6000 t/d of water, that brings back to the extraction process around 90 t of sugar and 30 t of impurities. Both components are undesirable, since their presence lowers the sugar extraction efficiency as well as juice purity and overall productivity.

Reverse osmosis (RO) could provide permeate consisting almost of pure water to be used in the extraction unit and concentrate of all components, which can be sent directly to the low grade crystallisation stage by-passing the high grade crystallisation stage and can be promptly eliminated in molasses. As can be seen from the upper part of Fig. 2, experiments were conducted on the two-stage RO system with pre-filtration step. It was used high retention RO membrane OSMO 411T-MS10 in spiral wound module 4×40”, NaCl retention 99%, at 3 MPa and 60°C.

Nowadays when NF membranes are available, another flow-sheet, different from that proposed by Bogliolo [18], could be considered, where the first stage of RO is replaced by NF unit as it is indicated in the bottom part of the Fig. 2. It will result in two retentate schemes, the NF permeate being the input for RO stage. Then RO permeate will go back to the extraction stage. NF retentate, containing sugar and high molecular impurities (proteins, high molecular colorants, multivalent salts) will go directly to the low grade crystallisation, while RO retentate containing mostly low molecular inorganic non-sugars will be wasted. In

this way the amount of molasses could be reduced comparing to the scheme in [18], since monovalent cations Na^+ and K^+ which easily pass through the NF membrane and will be wasted via RO retentate are responsible most of all for production of molasses, as can be seen from the Table 1 [19].

2.4. NF treatment of waste brine from sugar decolorizing resin regeneration

The last case discussed in this paper is NF recovery of sodium chloride from waste brine from the regeneration of anion exchange resin (See item 4 in the Fig. 1). Resins are used to remove high -molecular colorants like melanins, melanoidines, caramels and polyphenols from sugar liquor. Typical molar mass of colorants ranges from 500 to 20 000 D with the maximum 5000–20 000 D. The colorants are at first adsorbed onto the resins and finally released from the exhausted resin into alkaline 100 g/l sodium chloride solution producing a stream characterised by high salinity, high amount of coloured organic matter and high COD (13 000 mg/l), which represents a serious pollution problem. First tests with UF membranes result only in 45% reduction in organic matter and UF membranes were found not to be tight enough.

Again the advent of NF membranes brought an elegant solution, since the NaCl retention of NF membranes is as low as 10–50%, while organic compounds retention was reported by Wadley et al. [20] in 1995 to be in the range of 80–97% on Kiryat Weizmann (today Koch) NF membranes SelRO MPT-30 or MPT-31. It resulted in 30% reduction in effluent volume and 60% reduction in salt consumption. In 1997 Cartier et al. [21] refers, that with spiral wound membranes Desal 5.1 and Desal 5.2 (today Osmonics) and Filmtec NF4S membrane (Dow) it was possible to reach even 89% reduction in water consumption and 74% reduction in salt consumption. Moreover, the volume of toxic wastes discharged from sugar refinery was lowered.

Table 1
Melassogenic coefficients of different compounds

NaCl	2.58
KCl	2.48
NaOH	4.61
$CaCl_2$	0.56

3. Experimental studies

3.1. Ultra and microfiltration tests

The aim of these experiments with MF/UF of raw juice originating from the sugar refineries is discovering conditions, which enable to produce crystallisation without the prerequisite of epuration.

3.1.1. Properties of raw juice used

Raw juice samples taken from different extractors in sugar refineries during sugar beet campaign 1998 were analysed. Raw juice concentrate prepared by evaporating of fresh juice (falling film evaporator Armfield, UK) was also measured particularly to create the possibility of performing the newly designed technological process not only during a season but the whole year. Even a filtration area may be reduced in an all-year-round operation. This may possibly reduce the increased capital expenditure on membrane filtration. The properties of the raw juice and the concentrate are summarised in Table 2.

3.1.2. Methodology

The filtration kinetics (i.e. the permeate flux velocity dependence on time, temperature and operating pressure) was particularly observed. The permeate and retentate samples were measured in terms of colour, content of refractometric dry substance (RDS), content of sucrose, conductivity, contents of invert sugar (i.e. the contents of glucose and fructose produced due to the sucrose inversion) and lactic and acetic acids.

3.1.3. Filtration plant

The filtration unit is a pilot plant type made by the French firm T.I.A. Bollene (See Fig. 3) for scientific research as well as operation experiments, since it is equipped with standard filtration

Table 2
Properties of original raw juice in the campaign of 1998 and diluted concentrate of raw juice

	Original raw juice	Diluted concentraten
RDS (%)	14.2–16.8	14.9
Purity Q(%)	88.5–91.0	85.8
Colour substances Cb (cm^2/kg)	4500–7250	2550
pH	5.9–6.4	6.12
Acetic acid (% of RDS)		0.45
Lactic acid (% of RDS)	0.08	0.30

modules used in the industry. The device is equipped with two ceramic membranes Membralox, having filtration area of 2×0.2 m^2, porosity 20–100 nm for ultrafiltration and 0.2–5 μm for microfiltration. The limits within which experiments may be done are; temperatures up to 85°C, pressures up to 0.6 MPa with pH in a large range of 0.5–13.5. The pilot plant is designed for the following research applications in food technologies: milk and whey thickening, clarifying and thickening of fruit and sugar juices, protein suspensions, filtration in wine-growing and brewing industry, water treatment (removing of micro-organisms, sterilisation), waste water purification (separation of oils etc.).

3.1.4. Results and discussion

The filtered samples were treated with cross-flow micro- and ultrafiltration on ceramic membranes having porosity of 20 nm. Filtration was proceeded in the mode of retentate and permeate recycling with constant membrane pressure difference 0.1 or 0.2 MPa and constant working temperature 30, 50 and 60°C. Permeate and retentate samples were regularly taken in 5–30 min intervals for 3–10 h. Permeate flux was measured in the same intervals for the determination of filtration kinetics.

The values of dry substance and sucrose content, purity and colour were measured during the filtration. Conductivity and the contents of lactic acid and acetic acids were determined in the first and the last samples of diluted concentrate to compare these values with those of the retentate. The changes in the juice composition were observed in the analyses particularly focused on the sucrose, invert sugar (i.e. fructose and glucose) and lactic acid contents.

An interesting result was achieved in increasing the juice purity:

1. at 30°C: from the initial value 90.1% to the final value 91.5–92.5% of the original raw juice (See Fig. 4),

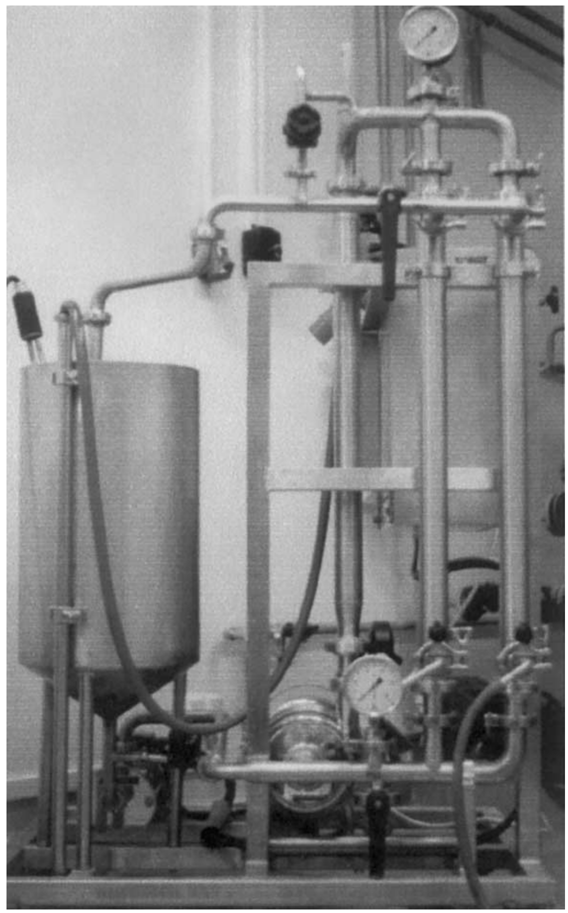

Fig. 3. T.I.A. micro- and ultrafiltration unit.

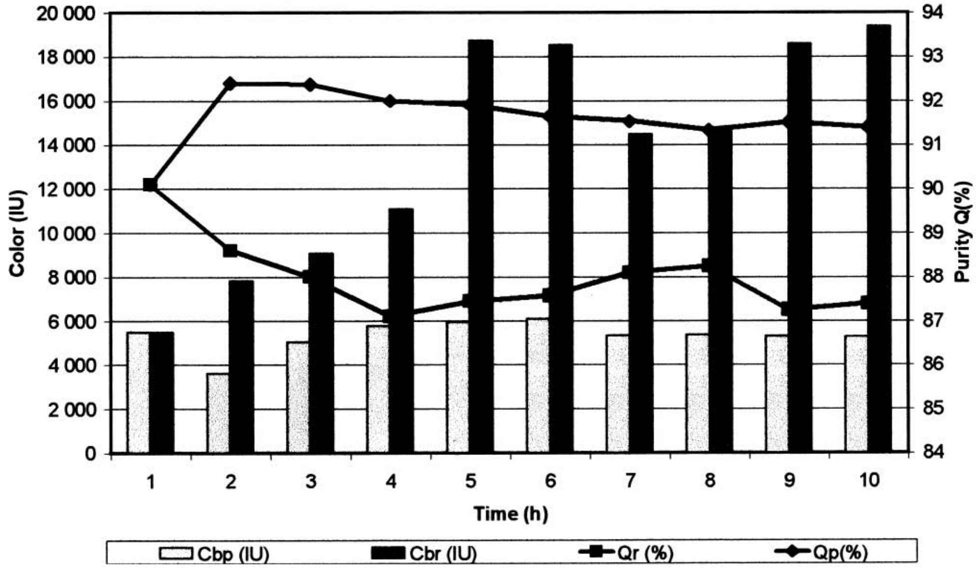

Fig. 4. Purity and colour of permeate and retentate of filtered raw juice. Conditions; temperature 30°C, pressure difference 0.1 Mpa, Legend; cb_P permeate colour, cb_R retentate colour, Q_P permeate purity, Q_R retentate purity.

2. at 50°C: from the initial value 88.2% to the final value 89.5–90.5% of the original raw juice.

The average value of impurities rejection [$R = 1 - (100 - Q_P)/(100 - Q_R)$] was calculated: 0.33 at temperature 30°C and 0.23 at 50°C.

Another significant result is the decrease of colour impurity content in the original raw juice by 50–70% and 50–60% in the concentrate. Colour matter rejection R, where $R = 1 - cb_P/cb_R$, achieved an average value 0.61 at temperature 30°C and 0.55 at 50°C. During other micro- and ultrafiltration of different raw juices and diluted concentrate this rejection varied in interval from 0.49 to 0.62. The turbidity (colloid content) fell to less than 1% of its initial value. These effects are necessary for further treatment of permeate to white sugar.

In addition, permeate can be a subject for the direct crystallisation to produce the final product after its thickening. The retentate purity decreased to 87–88% at 30°C and to 86–87% at 50°C. A similar effect was found by microfiltration of all used juices and diluted concentrate. A number of substances (particularly nitrogen compounds, proteins, polysaccharides) gathered in the retentate make this product a high-quality feed, which will improve the economic balance of costs and production when membrane filtration is applied.

3.2. Nanofiltration tests

The objective of this part of the work was to carry out preliminary experiments revealing if recent NF membranes can successfully treat sugar juices. If rejection are high enough, clean sugar solution could be obtained after two-step process involving UF removing of high molecular nonsugars and NF, where low molecular colorants and inorganic substances can be separated into permeate.

3.2.1. Material

Microfiltered juice from the Czech sugar beet factory Uzice, concentrated and subsequently diluted to 17% of RDS was used for all experiments. Only nanofiltration test No.2 (See Fig. 6) ran for comparison with unfiltered juice.

3.2.2. Methods and conditions

Tests were performed at very low tangential velocity 0.7 cm/s and pressure difference only 0.15

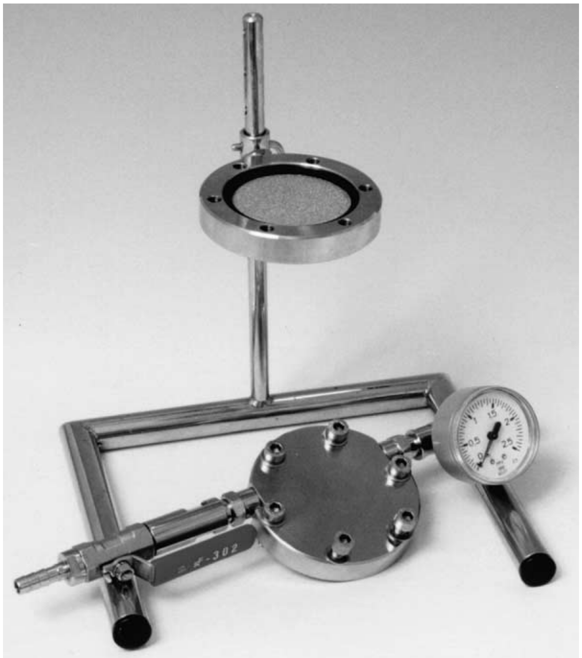

Fig. 5. Dynamic nanofiltration cell Micropur.

MPa, so that we rather speak about low-pressure static experiments. The permeate was collected and measured, the retentate recycled under ambient temperature 25°C back to the feed tank.

3.2.3. Equipment

Special high-pressure dynamic cell was designed for NF experiments (See Fig. 5). The unit with adjustable tangential speed 0–3 m/s enables to perform tests with variety of flat NF membranes. Circular motion of liquid in an open channel with no spacer above the membrane surface simulates process conditions close to the industrial disc-tube modules, which may be a good full-scale alternative for this application [22]. The channel profile was constructed in the way that the tangential velocity on individual radial co-ordinate is always the same.

3.2.4. Membrane used

Designation and properties of used membranes are compiled in Table 3.

3.2.5. Results and discussion

Permeate flux of filtered and unfiltered raw juice from first experiments for different types of NF membranes is shown in the Fig. 6. From the curves it is obvious that the behaviour was very sensitive to pressure discrepancies in the retentate flow. Some improvements in the retentate flow resulted in the washing of the membrane surface and subsequently in higher permeate flux (See experiments No.2 and 3, Fig. 6). Although the curves exhibit shapes which were expected, the results of analyses do not allow to make quantitative conclusions.

NF membrane from more open side of the spectrum which have desirable small NaCl rejection exhibited no separation effect for sugar and inorganic non-sugars under given conditions, what was expected regarding the data in Table 3. Membranes indicated as having cut-offs of 150–300 D or $MgSO_4$ (MW 120) retention 96–98% exhibited retention for sucrose only 50% in spite of larger molecule of sucrose (MW 342) while retention for non sugars ranged from 15–30%.

Table 3
Rejection (R) of salts on polymeric membranes published by producers

Membrane	R NaCl (%)	R $MgSO_4$ (%)	R Sucrose (%)	Curve No. (See Fig. 6)
Hydronautics Nitto Denko 7410*	15	9	5	1, 2
Hydronautics NittoDenko 74l0*	51	32	36	3, 6
Osmonics Desal DL		96		8
Osmonics Desal DK		98		No permeate
Hydranautics ESNA**	85			No permeate
Hydranantics PVD1				No permeate

* Test condition: 0.5% solution, 25°C, 1 Mpa.
** Test condition: 500 mg/l solution, 25°C, 0.52 Mpa.

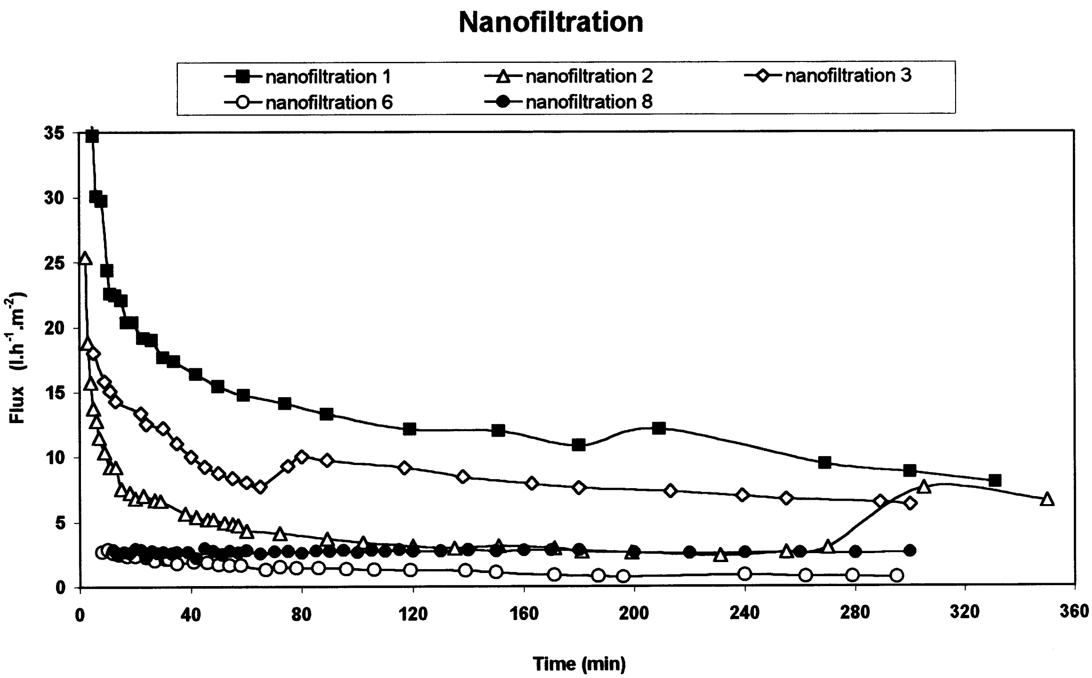

Fig. 6. Permeate fluxes during nanofiltration of sugar juice. Legend; 1 filtered juice membrane 7410, 2 nonfiltered juice membrane 7410, 3 nonfiltered juice membrane 7450 B, 6 nonfiltered juice membrane 7450 B, 8 nonfiltered juice membrane Desal DL.

Membranes from more dense side of the spectrum gave already no permeate due to the osmotic pressure barrier. The experimental work needs to be continued from this point view.

4. Conclusion

4.1. Ultra- and microfiltration tests

The obtained results showed that the juice treated with micro- and ultrafiltration achieved such quality that directs crystallisation was possible. The crystals achieved from the filtered juice are of subsequently better quality (compared with nontreated juice) particularly as regards the contents of colour matters and colloids; however, the results require a verification under industrial conditions because the quality and content of impurities in the juices varied a lot during the campaign.

4.2. Nanofiltration tests

Preliminary test showed that proper membrane for NF of raw juice should be sought on the dense side of the NF membrane spectrum where some tested membranes exhibited higher retention for sucrose than for non-sugars. Higher pressure and tangential velocities than used in this work should be applied.

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